REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. REPORT DATE *(DD-MM-YYYY)*	2. REPORT TYPE	3. DATES COVERED *(From - To)*
	Final Report	01 February 2003 - 31 December 2005

4. TITLE AND SUBTITLE

Synthesis and Microstructural Design of Oxide Fibers

5a. CONTRACT NUMBER

5b. GRANT NUMBER

F49620-03-1-0082

5c. PROGRAM ELEMENT NUMBER

6. AUTHOR(S)

Professor Waltraud M. Kriven

5d. PROJECT NUMBER

5e. TASK NUMBER

5f. WORK UNIT NUMBER

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

University of Illinois, Urbana-Champaign
Department of Materials Science and Engineering
Urbana IL 61801

8. PERFORMING ORGANIZATION REPORT NUMBER

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)

USAF/AFRL
AFOSR
875 N. Randolph Street
Arlington VA 22203
Dr Joan Fuller/NA

SPONSOR/MONITOR'S ACRONYM(S)

AFRL-SR-AR-TR-06-0307

12. DISTRIBUTION/AVAILABILITY STATEMENT

Distribution Statement A. Approved for public release; distribution is unlimited.

13. SUPPLEMENTARY NOTES

14. ABSTRACT

Fine ceramic oxide fibers are widely used as reinforcements in composites for high temperature applications. The primary goal of this research effort was to investigate the growth of single crystal or textured or eutectic oxide fibers by heat treatment of polycrystalline or amorphous precursor fibers.

20060727332

15. SUBJECT TERMS

16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
a. REPORT	b. ABSTRACT	c. THIS PAGE			19b. TELEPHONE NUMBER *(Include area code)*
U	U	U	UU	6	

Synthesis and Microstructural Design of Oxide Fibers

F49620-03-01-0082

Wonki Yoon
Waltraud M. Kriven
Department of Materials Science and Engineering
University of Illinois, Urbana-Champaign

Abstract

Fine ceramic oxide fibers are widely used as reinforcements in composites for high temperature applications. The primary goal of this research effort was to investigate the growth of single crystal or textured or eutectic oxide fibers by heat treatment of polycrystalline or amorphous precursor fibers.

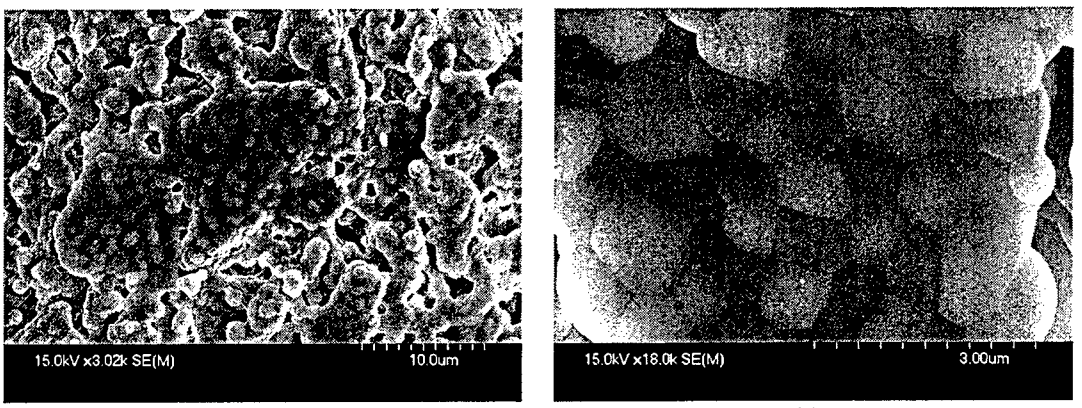
Mullite and alumina-YAG eutectic fibers were prepared as precursor fibers for the heat treatment. Mullite, alumina-YAG eutectic composition powders were synthesized by PVA-based, steric entrapment method. Oxide fiber was extruded with a Marksman fiber extruder. In addition, natural silk monofilaments from silkworm cocoons and commercial cotton fibers were infiltrated with mullite and alumina-YAG eutectic composition sols in order to obtain small diameter fibers. Different kinds of sols were prepared and the fibers were infiltrated and dried. The precursor fibers prepared were crystallized with our in-house quadrupole lamp furnace to obtain single crystal or textured or eutectic crystalline oxide fibers.

Alumina-YAG eutectic composition (50.67mol% of alumina and 49.33mol% of YAG) powder was prepared by the organic steric entrapment method. The heat treatments of the extruded fibers were carried out at up to 98% of furnace power with the quadrupole lamp furnace. As the power of the furnace increased, densification and grain growth of alumina and YAG phases occurred. When the fiber was heat treated above the melting temperature, the microstructure changed to a eutectic microstructure. It is expected to have high strength at high temperatures due to the interlocked microstructure.

Mullite whiskers were synthesized from sintered titania-doped mullite powder. The collected mullite whiskers were embedded in the mullite fiber for templated grain growth. The whiskers were aligned in the fiber length direction by extrusion. Elongated grain growth occurred with the embedded whisker samples heated in the quadrupole lamp furnace. The textured mullite microstructure and single crystalline mullite fiber could be achieved after further heat treatment. The microstructure of the single crystalline mullite fiber was examined with an optical microscope in transmitted polarized light, and with a transmission electron microscope. It was confirmed that the grown direction along the fiber length was the [001] direction of orthorhombic mullite.

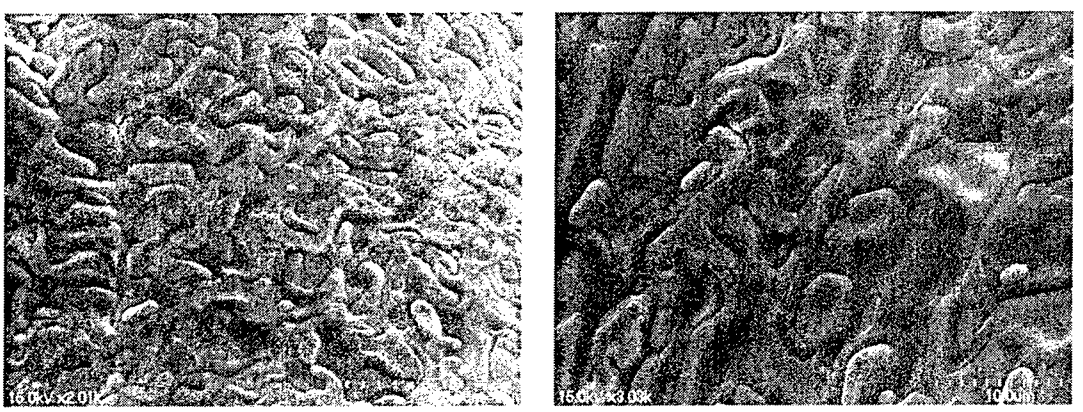
Research Objective

The objective of this study is to develop ceramic oxide fibers for reinforcement of the composites for high temperature use. The intrinsic brittleness of the ceramics can be modified by employing ceramic fibers. Mullite and yttrium aluminum garnet (YAG) were studied due to their superb high temperature properties such as creep. Polycrystalline fibers were crystallized into single or textured microstructures. Also the eutectic microstructure was prepared by directional solidification. By crystallization of polycrystalline or amorphous precursor fibers into single crystal or textured oxide ceramic fibers, the strength of fibers can be increased.



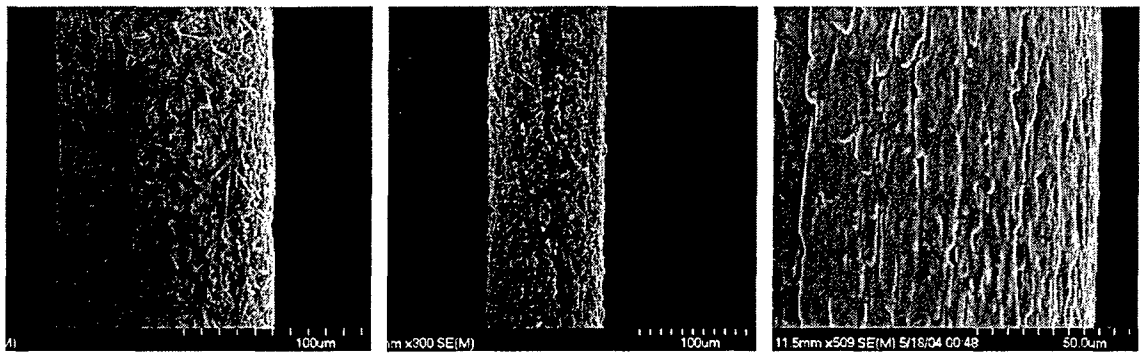
(a) (b)

Figure 1: (a) SEM micrograph of alumina-YAG eutectic composition fiber heat treated in $MoSi_2$ furnace at 1600°C for 2hours, (b) higher magnification image of (a).



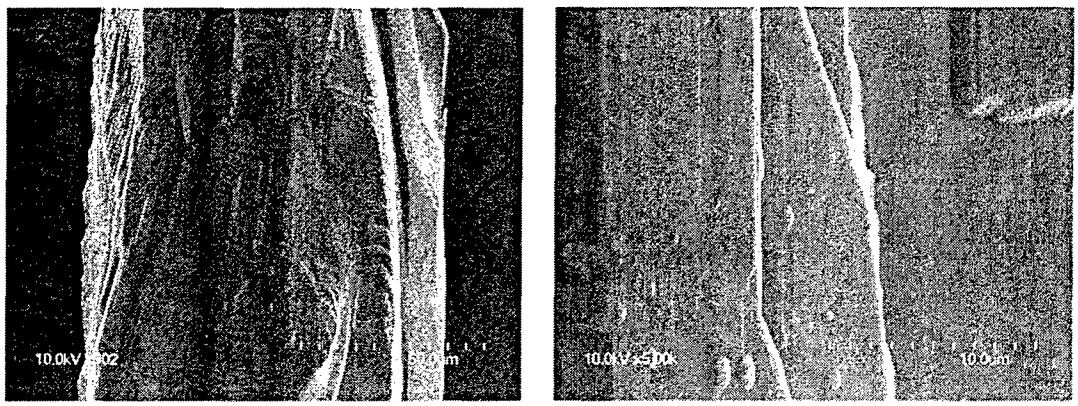
(a) (b)

Figure 2: (a) SEM micrograph of alumina-YAG eutectic microstructure heat treated in quadrupole lamp furnace at 98% of power(~2010°C), at a transverse rate of 0.01 mm/s (b) higher magnification image of (a)



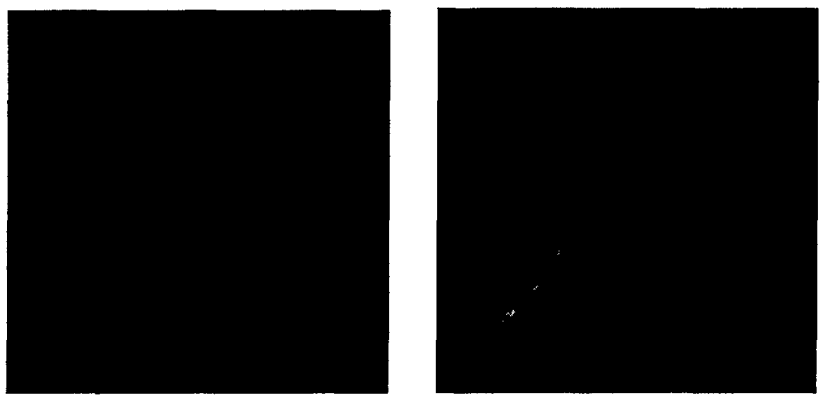
(a) (b) (c)

Figure 3: SEM micrographs of extruded 5 wt% mullite whisker-added, mullite fiber: (a) heat treated at 76% of power(~1780°C), at a transverse rate of 0.01 mm/s, (b) heat treated at 87% of power(~1830°C), at a transverse rate of 0.001 mm/s, and (c) heat treated at 80% of power(~1810°C), at a transverse rate of 0.001 mm/s.



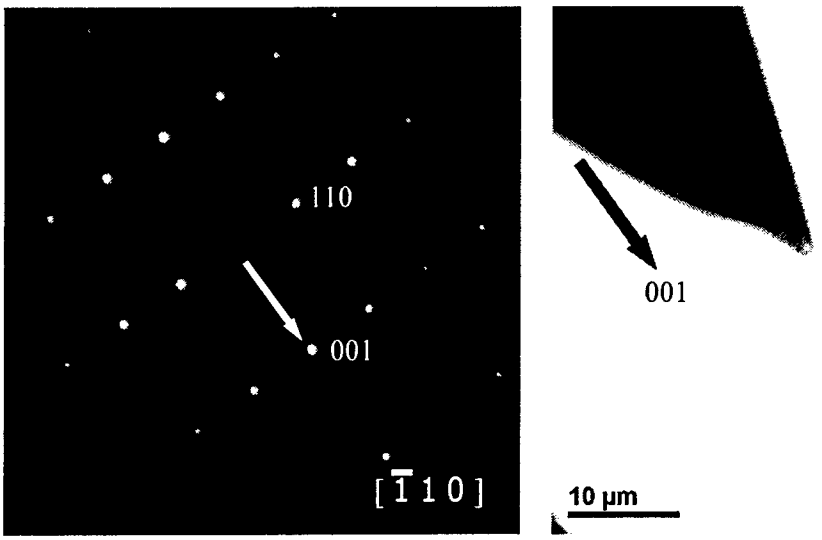
(a) (b)

Figure 4: (a) SEM micrographs of extruded 5 wt% whisker added mullite fiber heat treated 10 times at 85% of power(~1820°C), at a transverse rate of 0.001 mm/s , (b) high resolution of (a).



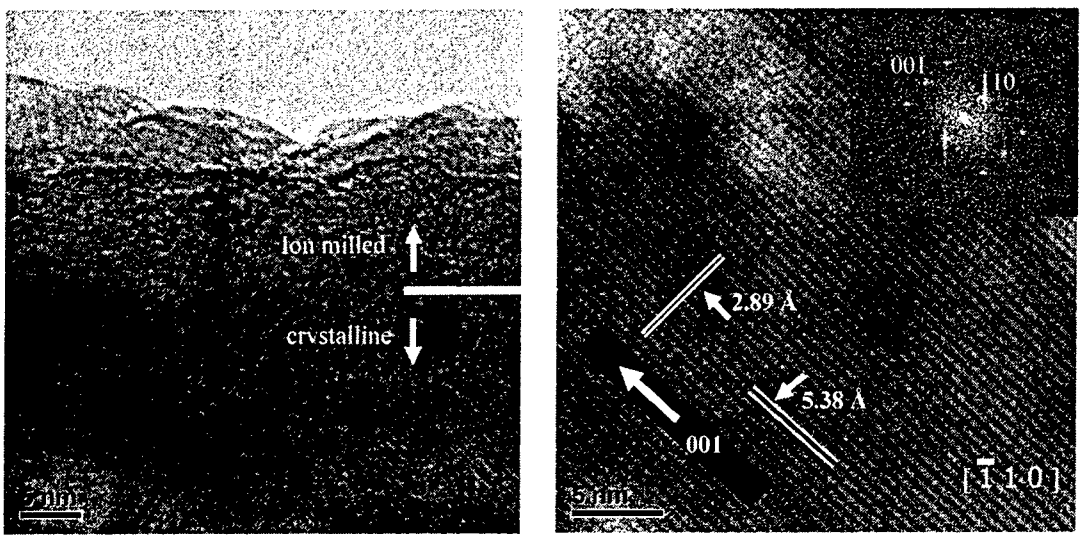
(a) (b)

Figure 5: Optical microscope image of whisker added mullite fiber heat treated 10 times at 85% of power(~1820°C), at a transverse rate of 0.001 mm/s, (a) total extinction, (b) optical microscope image in transmitted polarized light.



(a) (b)

Figure 6: (a) TEM diffraction image of whisker-added, mullite fiber heat-treated 10 times at 85% of power(~1820°C), at a transverse rate of 0.001 mm/s(1μm/s), (b) defocused direct beam in a diffraction pattern.



(a) (b)

Figure 7: (a) TEM micrographs of ion-milled mullite fiber heat treated 10 times at 85% of power(~1820°C), at a transverse rate of 0.001 mm/s , (b) high resolution of TEM image of mullite single crystalline fiber.

Acknowledgment/Disclaimer

This work was sponsored by the Air Force Office of Scientific Research, USAF, under grant/contract number F49620-03-01-0082. The views and conclusions contained herein are those of the authors and should not be interpreted as necessarily representing the official policies or endorsements, either expressed or implied, of the Air Force Office of Scientific Research or the U.S. Government.

References

1 A. R. Bunsell and M. –H. Berger, "Fine Ceramic Fibres," *Journal of the European Ceramic Society*, **20** [13] 2249-60(2000),

2 H. Schneider, K. Okada and J.A. Pask, "Mullite and Mullite ceramics," John Wiley, New York, NY, 1994,

3 A. Sayir and S. Farmer, "Directionally Solidified Mullite Fibers," pp. 11-21 in *Ceramic Matrix Composites-Advanced High Temperature Structural Materials*, Edited by R. A. Lowden, M. K. Ferber, J. R. Hellmann, K. K. Chawla, and S. G. DiPietro, *Materials Research Proceedings*, [365] (1995),

4 S. Hong and G. L. Messing, "Anisotropic Grain Growth in Diphasic-Gel-Drived Titania-Doped Mullite," *Journal of the American Ceramic Society*, **81** [5] 1269-77 (1998),

5 B. R. Johnson, W. M. Kriven, and J. Schneider, "Crystal Structure Development during Devitrification of Quenched Mullite," *Journal of the European Ceramic Society*, **21** 2541-2562 (2001),

6 W. M. Kriven and J. A. Pask, "Solid Solution Range and Microstructures of Melt-Grown Mullite," *Journal of the American Ceramic Society*, **66** [9] 649-54 (1983),

7 Ceramic Transactions Vol. **6**, "Mullite and Mullite Matrix Composites," Edited by S. Somiya, R. F. Davis and J. A. Pask, American Ceramic Society, Westerville, OH, 1990,

8 C. Scott, M. Kaliszewski, C. Greskovich and L. Levinson, "Conversion of Polycrystalline Al_2O_3 into Single Crystal Sapphire by Abnormal Grain Growth," *Journal of the American Ceramic Society*, **85** [5] 1275-80 (2002),

9 T. Mah, T. A. Parthasarathy and R. J. Kerans, " Processing, Microstructure, and Strength of Alumina-YAG Eutectic Polycrystals," *Journal of the American Ceramic Society*, **83** [8] 2088-90 (2000),

10 D. Park, J. Yang, and J. M. Collins, "Coarsening of Lamellar Microstructures in Directionally Solidified Yttrium Aluminate/Alumina Eutectic Fiber," *Journal of the American Ceramic Society*, **84** [12] 2991-96 (2001).

Personnel Supported

Wonki Yoon	Graduate Student, University of Illinois at Urbana-Champaign
Kerstin Jurkschat	Postdoc, University of Illinois at Urbana-Champaign

Publications

"Fabrication and Grain Growth in YAG and Mullite Fibers," W. M. Kriven, K. Jurkschat, B. R. Johnson, W. Yoon and C. Chiritescu, Ceramic Transactions vol. 153, (2003).

"Investigations on Growth of Textured and Single Crystal Oxide Fibers Using a Quadrupole Lamp Furnace," W. Yoon and W. M. Kriven, for the proceedings of the 29th International Conference on Advanced Ceramics and Composites, *submitted.*

Presentations

1. "Investigations on Growth of Textured and Single Crystal Oxide Fibers Using a Quadrupole Lamp Furnace", W. Yoon, C. Chiritescu and W.M. Kriven, 29th Annual Cocoa Beach Conference and Exposition on Advanced Ceramics and Composites, Cocoa Beach, Florida, Jan 2005.
2. "Quadrupole Lamp Furnace – An Excellent Tool for Conducting In-Situ High Temperature X-ray Diffraction", P. Sarin, K. Jurkschat, W. Yoon, A. J. Randolph and W. M. Kriven, Condensed Matter Physics Seminar (Invited), Brookhaven National Laboratory, Upton, New York, July 2004.
3. "Grain Growth and Texture Development in YAG and Mullite Fibers," K. Jurkschat, W. M. Kriven, W. Yoon and C. Chiritescu, 105th Annual Meeting and Exposition of the American Ceramic Society, Nashville, Tennessee, April 2003.
4. "Fabrication of YAG and Mullite Fibers," W. M. Kriven,* K. Jurkschat, W. Yoon and C. Chiritescu, 105th Annual Meeting and Exposition of the American Ceramic Society, Nashville, Tennessee, April 2003.
5. "Crystallization and TEM Microstructure of Oxide Fibers", W. M. Kriven, B. R. Johnson and W. Yoon, Annual Meeting of the Materials Research Society, Boston, Massachusetts, Dec 2001.